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A preliminary report on the crystal structure of phenacetin paracetamol, solasodine monohydrate and 4-Phenyl 5, 7-dihydroxy 6-isovaleryl 8-isopentnyl coumarin

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The crystal structure determination of a number of industrially important compounds have been taken up in order to elucidate their detailed stereochemistry and get an insight into their activities. This is of great interest in the developmental work being carried out in this laboratory. A preliminary report on the structural study of four of them is presented here.

Phenacetin

Phenacetin, the wellknown analgesic and antipyretic drug has the chemical formula in figure 1.

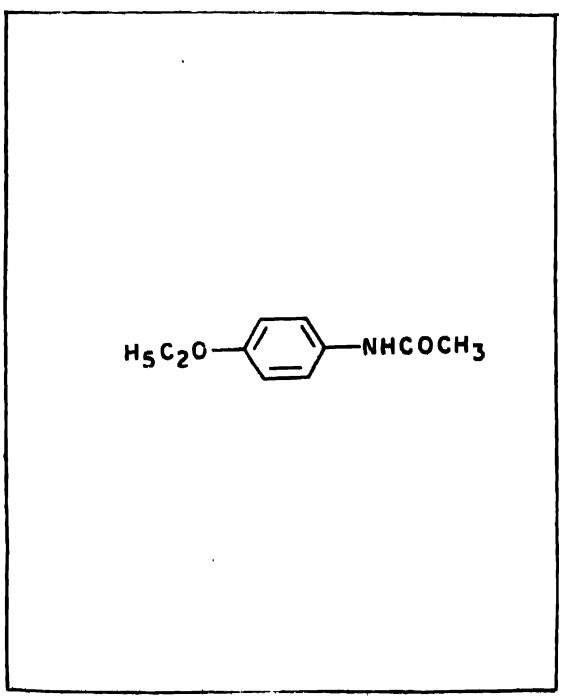

Fig. 1

It crystallizes as colourless rods elongated along *c*-axis in slow evaporation of an alcoholic solution of the compound.

Rotation and Weissenberg X-ray photographs taken about *b* and *c* axes showed that the crystals belong to the monoclinic system with $a = 13{\cdot}44$, $b = 9{\cdot}59$, $c = 7{\cdot}77$ Å and $\beta = 104{\cdot}3°$. The only systematic absences are for oko with k odd and hol with l odd, indicating that the space group is $P2_1/c$. The density of the crystal as determined by flotation method is 1·21, while that calculated for 4 molecules of $C_{10}H_{13}NO_2$ per unit cell is 1·22 g. cm^{-3}.

Complete three dimensional data have been collected using multiple-film equi-inclination Weissenberg technique with CuK_α radiation. The intensities of the spots have been estimated visually and corrected for Lorentz, polarization and spot size effects (Phillips 1954, 1956). They have then been brought to the same absolute scale by cross layer correlation and statistical method (Wilson 1942). The structure determination is in progress.

Paracetamol

This compound has similar drug activities as those of phenacetin. Its chemical formula is in figure 2.

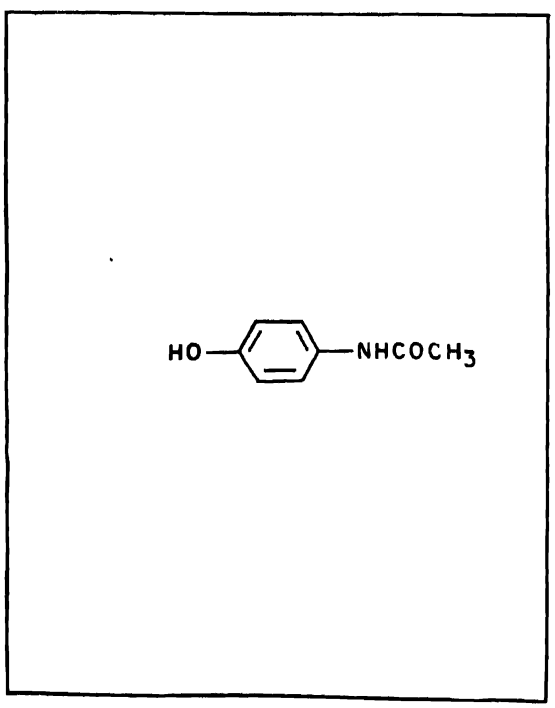

Fig. 2

The compound crystallizes as thin transparent plates from alcohol. X-ray analysis reveals that the crystals belong to the monoclinic system. The cell

constants are $a = 12{\cdot}43$, $b = 9{\cdot}49$, $c = 7{\cdot}21$ Å and $\beta = 109{\cdot}1°$. The observed systematic absences are for oko with k odd and hol with $h+l$ odd. The space group is therefore $P2_{1/n}$. The observed density of the crystal is 1.25 g. cm^{-3}, the same as that calculated for 4 molecules of $C_8H_9NO_2$ per unit cell. Collection of three dimensional intensity data for this compound is in progress.

Solasodine monohydrate

This is an alkaloid extracted from the berries of *Solanum Khasianum*. It is of great industrial and medicinal value, since a number of hormones like progesterone, estrone and 17α-ethynyl-19-nortestosterone (oral contraceptives), as well as cortisone and cortisone acetates (used in various ailments) are derived from this. The chemical structure of this compound is shown in figure 3.

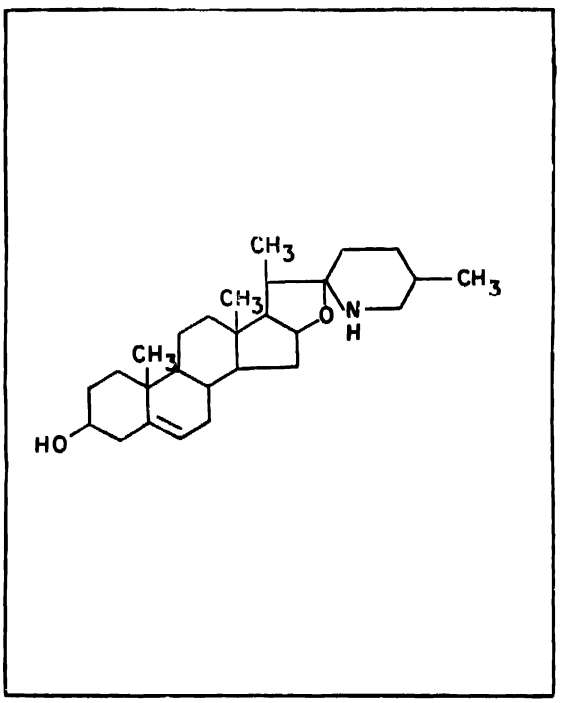

Fig. 3

It is very similar to that of tomatodine from which, however, it remarkably varies in activity. Since the full sterochemistry of tomatodine has been elucidated by X-ray methods (Konnard, Sansaverino & Rollett 1967, Hoehne, Ripperger & Schreiber 1967) it will be of interest to find the structure of solasodine as well and compare the two structures for a possible explanation of the activity.

X-ray photographs reveal that the crystals of this compound are in the orthorhombic system. The cell dimensions are $a = 7{\cdot}64$, $b = 9{\cdot}57$, $c = 16{\cdot}73$ Å.

Systematic absences for hoo with h odd, oko with k odd and ool with l odd were observed. The space group is $P2_12_12_1$, with one molecule per asymmetric unit, as confirmed by density determination by flotation method. Intensity data collection are in progress.

4-Phenyl 5,7-dihydroxy 6-isovalery 8-isopentnyl coumarin

This compound forms a part of the essential oil derived from cinnamon bark. The chemical structure of this compound as elucidated in this laboratory (Shivakumar, Mathur & Gopinath 1974) is given in figure 4.

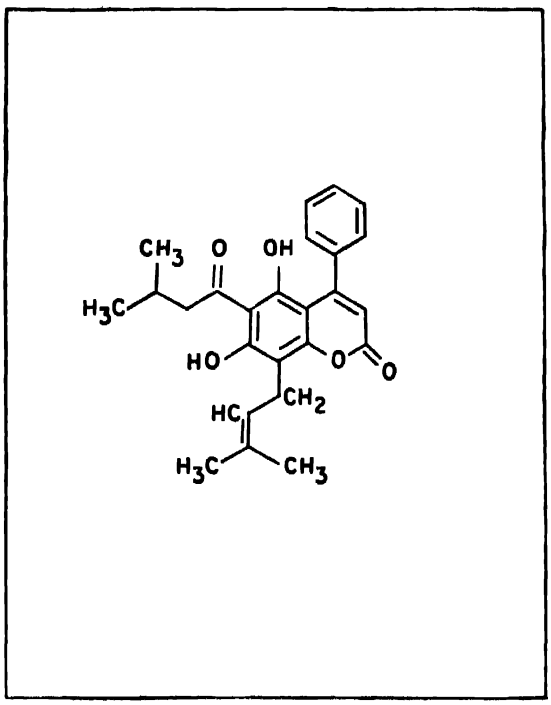

Fig. 4

The crystal grows in orthorhombic system with cell dimensions as $a = 15{\cdot}42$, $b = 16{\cdot}08$, $c = 20{\cdot}87$Å. The space group is $P2_12_12_1$, (hoo with h odd, oko with k odd and ol with l odd reflections are absent). Further work on this compound is not contemplated at present.

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